Engineering and Technology Journal
Journal homepage: https://etj.uotechnology.edu.iq

Effect of Subcritical Treatment (Tempering) on Hardness and Wear of High Chromium White Cast Iron

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HIGHLIGHTS

- The effects of tempering treatments on the service life of grinding balls were analyzed.
- The relationship between hardness, abrasive wear, and microstructure HCWCI was determined.
- The role played by matrix and carbides in improving the hardness and wear resistance was investigated.

ARTICLE INFO

Handling editor: Akram R. Jabur

Keywords:
HCWCI
grinding
tempering
hardness
wear

ABSTRACT

High chromium white cast iron (HCWCI) is used in mining, crushing plants, as mill liners, and in the manufacture of grinding balls. The grinding ball is the essential element in fine fragmentation. It must have an excellent life to counter extreme wear and impact conditions that it undergoes. Hence it is important to improve its mechanical properties. This study aims to find the effect of tempering temperature on microstructure, hardness, and abrasive wear of High Chromium White Cast Iron used in the manufacture of grinding balls. In this study, the balls in high chromium white cast iron with 12 to 17% Cr, of 50mm and 70 mm in diameter were austenitized at 950°C for 45 min and 55 min for the balls of 50 and 70mm then were quenched in forced air. The balls were tempered at 250°C, 400°C, and 600°C for 120 min and 180 min for both diameters 50 and 70mm respectively, and cooled in the furnace. The results showed that tempering practiced at 250°C and 400°C gives an excellent hardness than balls austenitized at 950°C. It is about 60 HRC and it drops to less than 50 HRC for the tempering at 600°C. This tempering causes a significant mass loss (short life) but tempering at 250°C and 400°C decreases this loss by up to 1.6%. The results of XRD showed the presence of the martensite and carbides type M_7C_3 after tempering at 250°C and a ferritic matrix after tempering at 600°C.

1. Introduction

High chromium white cast irons, which contain 12 to 30% Cr and 2.9 to 3.6% C, provide numerous and significant performances clearly superior to that of other cast irons for needed high abrasion wear resistance applications. These cast irons are easily identified by their hard eutectic carbides of $(Cr, Fe)_7C_3$ or M_7C_3 and austenitic dendritic matrix obtained in as-cast condition [1-3]. The size and morphology of these carbides control the wear resistance and toughness [4]. This initial morphology can be notably modified by the utilization of various critical and subcritical heat treatments, the purpose of which is the precipitation of secondary carbides and the transformation of the destabilized austenite into more desirable morphologies, such as martensite [5-7].

Chrome cast irons are generally tempered after the quench hardening treatment. The quenching heat treatment is usually conducted at 900–1050°C for 1–6 hr. The tempering heat treatment usually follows the quenching treatment and is carried out at 200–600°C for 2–6 hr, with the purpose of further precipitation of secondary carbides and elimination of any retained austenitic phase after the destabilization treatment [8, 9]. These treatments (destabilization) have been reported in many research papers. However, the research on sub-critical heat treatment is quite limited [10]. Generally, the carbides and matrix of HCWCI, formed during the heat treatments are affected by chemical composition and process parameters during heat treatment, as we will see in what follows.

K.M. Ibrahim and M.M. Ibrahim [11] studied the effects of heat treatment on the microstructure and mechanical properties of high chromium white cast iron alloyed with titanium. The austenitization heat treatment with temperatures of 980°C and also 1150°C for 1 hour followed by tempering at 260°C for two hours has been accomplished, and the effect of these processes on (wear resistance/impact toughness) combination was reported. The results clarify that the maximum tensile strengths were

http://doi.org/10.30684/etj.2023.137551.1353
Received 28 December 2022; Accepted 29 January 2023; Available online 14 February 2023
2412-0758/University of Technology-Iraq, Baghdad, Iraq

achieved for the (1.31%Ti) irons; also, maximum impact toughness was obtained for the irons without Ti addition. The wear resistance results were higher for the samples austenitized at 980°C in comparison to the irons treated at 1150°C. For both treatments, optimum wear resistance was obtained with 1.3% Ti.

Kosasu et al. [7] studied the effect of silicon content on the variation of hardness and retained austenite during subcritical heat treatment of the 16wt% Cr cast iron. It is found that in the sub-critically heat-treated state, the hardness increased first and subsequently decreased as the holding temperature increased. The degree of an increase in hardness becomes small with the addition of Si up to 1.5%. The volume fraction of retained austenite decreased remarkably when the holding temperature increased over 823 K under the same holding time.

In the reference [10] the relationships between the subcritical heat treatment hardness, the volume fraction of retained austenite ($V\gamma$), and abrasive wear resistance of hypoeutectic 16% Cr-2% Mo cast irons with 0-3%V were investigated. It is found that the subcritical heat treatment can improve the hardness and wear resistance. The maximum hardness in the subcritical heat treatment was obtained in the specimen with 3% V. The highest wear resistance was obtained in the specimen in which the matrix consisted of martensite and austenite together with secondary carbides, and the lowest wear resistance was obtained in specimens with secondary carbides and ferrite in the matrix. The $V\gamma$ decreased greatly when the holding temperature was elevated over 823 K.

Abdel-Aziz [4] observed the effect of changing the chemical composition and heat treatments on the microstructure and mechanical properties of high chromium white cast iron. The results revealed marked improvements in mechanical properties and wear performance by adding carbide-forming elements. On the other hand, the destabilization heat treatment is employed to obtain the martensitic structure for improving the toughness and abrasion resistance of these irons. In addition, the subcritical (tempering) heat treatments following the hardening are commonly utilized to relieve internal stresses and to control the final hardness before service. Wang [12] studied the behavior of 16Cr-1Mo-1Cu white cast iron during subcritical treatment at 853 K (580°C) for different time intervals of processing. A sharp hardness increase was observed between 2 and 10 hr, followed by a smooth drop for the next 6 hr. Above 16 hr of processing, a slight increase was observed. These hardness fluctuations were associated with the different microstructural features.

Karantzalis et al. [9] studied the effects of subcritical heat treatments on the microstructure of a high chromium cast iron. The specimens were subjected to austenitization heat treatment at 850°C followed by subcritical heat treatments at 350, 450, 550, 650, 750, and 850°C for 30, 60, 120, 180, 240, 360, 480, 600, 720, 840, 960, 1080, 1200, 1800, 1920, and 2040 min followed by cooling in air. The phase transformations and microstructural observations were reported.

At both temperatures of 350 and 450 °C, no precipitation of secondary carbides was observed, and the overall microstructure resembles that of the as-cast condition. At 550 °C, hardness values increased slightly compared to the as-cast values. No evidence of secondary carbide formation was observed. At 650 and 750 °C, extensive to complete transformation to pearlite-ferrite structures has occurred. Some evidence of secondary carbide precipitation especially for prolonged treatment periods was not adequate to obstruct the hardness decrease due to the dominating effect of pearlitic-ferritic formation. At 850 °C, secondary carbide precipitation and martensite formation lead to high hardness values.

Simple techniques for measuring wear can be used to assess the amount of material removed [13]:

- Weighing: weighing of the sample before and after the friction test using a precision balance;
- Dimension Measurement: Measurement of variation of sample length before and after the friction test, in the case of a uniformly distributed wear;
- Worn volume measurement: Topography measurement before and after the test to assess the volume removed to create the wear track.

The first technique was applied by several researchers on marked balls placed in a laboratory ball mill with a group of standard balls of known quality. An "abrasion factor" or relative rate of wear was calculated [14, 15].

Laboratory ball mills are used by several research teams as another alternative to simulate the severe abrasion conditions encountered in industrial ball mills. They give accurate predictions of the service life of wear-resistant alloys in grinding applications. These tests are inexpensive and less time-consuming than the tests in industrial mills. The first attempts to develop these tests date from the 1940s [16, 17].

The grinding ball is widely used in fragmentation industries and in particular the cement industry. It is manufactured by the Algerian Foundries (ALFET - Tiaret) in high chromium white cast iron with 12 to 17% Cr. It undergoes abrasion wear that result of friction between many surfaces (rock, crusher shielding, and balls between them), between which a sliding contact occurs, and causes a metal wrenching and mechanical disintegration of these surfaces [18].

Only a limited amount of study has been focused on the direct application of subcritical heat treatments of high-chromium white cast irons [9]. The present research work attempts to clarify the effect of different tempering heat treatments on microstructure, hardness (HRC), and wear resistance of high chromium white cast irons. The scope of the present study is that the effect of subcritical treatments is carried out on real parts (balls of 50 and 70mm in diameter), which makes it possible to clarify the effect of the size of the balls on the hardness at the surface and in center of the ball (hardenability) and on the rate of wear of these balls.

2. Materials and Methods

2.1 Materials

The balls as-cast of 50 and 70 mm in diameter are procured from Tiaret Algerian foundries (ALFET) in Algeria. Their chemical compositions obtained by using analysis of X-rays fluorescence, are shown in Table 1.

Table 1: Chemical compositions (wt%) of high-chromium cast iron

C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Fe
2.87	0.56	0.6	0.06	0.086	14.0	0.7	0.33	0.15	Bal

2.2 Heat Treatments

Heat treatment of balls 50 and 70 mm in diameter was conducted in a muffle electric furnace with silicon carbide rod heaters. After austenitization at 950°C for 45 min and 55 min for 50 and 70 mm balls respectively, specimens were quenched in forced air.

The balls were tempered at 250°C, 400°C, and 600°C. The dwell time of the balls 50mm in diameter is 2 hrs and that of the balls 70mm in diameter is 3 hrs. They were cooled in a furnace. The cooling rate of the balls is about 100°C/hr. All heat treatment processes are summarized in Table 2.

Table 2: Heat treatment processes

Ball diameter (mm)	50			70		
Austenitizing temperature (°C)	950°C					
Dwell time at austenitizing temperature (DtQ, min)	45			55		
Cooling after austenitization	Forced air cooling					
Tempering temperatures (T_T,°C)	250	400	600	250	400	600
Dwell time at tempering temperature (Dtt, min)	120			180		
Cooling after tempering	Cooling in furnace					

The procedure of quenching at 950°C followed by tempering at 250°C, 400°C and 600°C is illustrated in Figure 1.

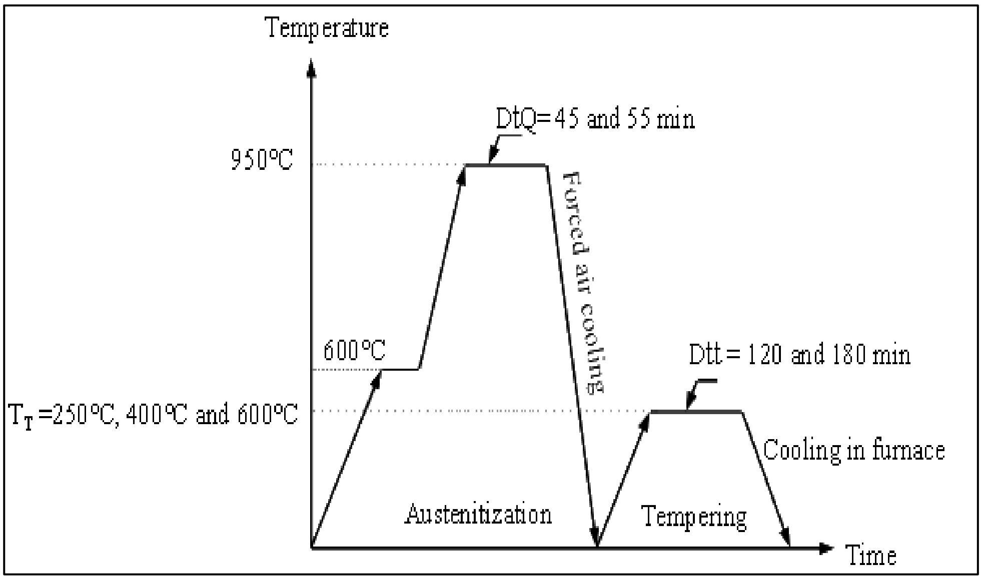


Figure 1: Austenitization and tempering procedure

2.3 Microstructure Investigations

The samples for metallographic examination were polished on a series of four (120, 240, 400, and 600) grit papers (European standards FEPA.P) and using very fine alumina suspended in water (type: DURMAX, fineness: 24 hrs). The specimens were etched with 4% initial solution and observed under an optical microscope ranging from 250 X to 500 X magnifications. The different phases which are present on samples for different tempering treatments were investigated using X-ray diffraction. Diffraction patterns were recorded in a Cu-anode X-ray tube using an X'PERT PRO MRD diffractometer in the range of angles 2θ = 30 – 100° in order to improve the signal-to-noise ratio, the time for collection of intensity was 40 sec with an angular step of 0.04°. X'Pert High Score software with a database of ICDD-PDF2 was used for phase identification.

2.4 Hardness Tests

Rockwell HRC hardness measurements are made on the smooth and flat surfaces of balls. The hardness is measured from the outer surface to the middle of the ball and this is by a progressive decrease in the thickness of the ball by 5 mm, 10 mm, 15 mm ...etc. and for an average of 5 measurements per surface Figure 2.

These tests were carried out using a hardness tester of the HP250 type (220 V / 50 HZ / 6 A) equipped with a conical diamond indenter with an angle at the top of 120° and rounded at its top (R = 0.2 mm).

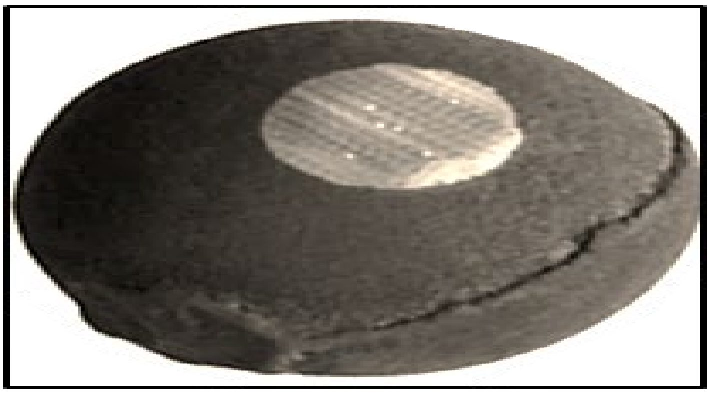


Figure 2: Rockwell HRC hardness measurement

2.5 Wear Tests

The exact evaluation of the service conditions in which wear occurs is very difficult to achieve and large-scale wear tests require considerable expense and an extremely long time. Moreover, most results found in the literature regarding wear resistance for white cast irons and steels were obtained by laboratory tests [14, 16].

The wear laboratory tests conducted during this study are carried out according to the first technique mentioned in the introduction.

The wear of the heat-treated balls is estimated, in this work, by the cumulative mass loss in (%), obtained by weighing before and after the wear test using a 0.1 g precision balance. These balls are put inside the drum of Figure 3, with a diameter of D=1.2 m for two hrs. This drum is animated with a uniform rotation motion of 28 rpm. The filling rate was 30%.

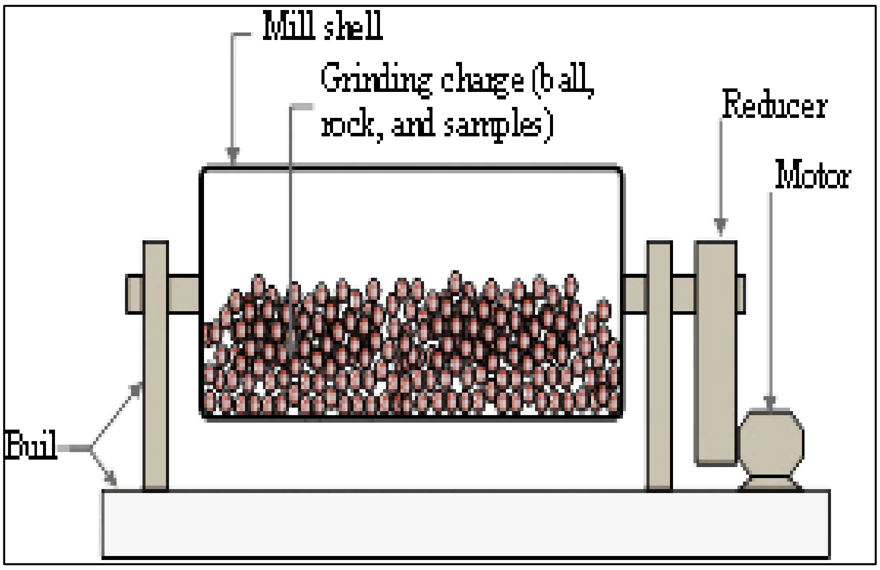


Figure 3: Experimental device for measuring balls wear

3. Results and Discussion

3.1 Rockwell Bulk Hardness

Tables 3 and 4 gives the hardness measured from the surface of the balls of 50 and 70 mm in diameter to their center, as cast, austenitized at 950°C and tempered at 250°C, 400°C, and 600°C.

Table 3: Hardness measured from the surface of the ball of 50 mm in diameter to its center

	Condition				
Distance from the surface (mm)	**As-cast**	**Quenching 950°C**	**Tempering 250°C**	**Tempering 400°C**	**Tempering 600°C**
5	48.66 HRC	63.66	62.8	62	45
10	48	64.4 HRC	64.07	62	45.6
15	47	62	60.6	61	42.3
20	46	61.25	61	60.56	40.5
25	45.66	60.42	60.52	60.1	40.5

Table 4: Hardness measured from the surface of the ball of 70 mm in diameter to its center

	Condition				
Distance from the surface (mm)	**As-cast**	**Quenching 950°C**	**Tempering 250°C**	**Tempering 400°C**	**Tempering 600°C**
5	47 HRC	65	64.3	61.4	48
10	45.6	64.36	63.66	61.2	45
15	45.25	64	62.6	59.7	44.1
25	45	62.6	62.15	59.2	43.6
30	44.8	61.65	61.2	58.4	43
35	44.63	60.25	59.8	57.65	43.1

The difference between the hardness of the surface and center is more significant for balls 70 mm in diameter than for balls 50 mm in diameter.

A comparison of the average bulk hardness of balls 50 and 70 mm in diameter tempering at 250, 400, and 600°C is given in Figure 4. The highest hardness is obtained for tempering at 250°C and 400°C; it is around HRC60 for balls of both diameters. It drops to less HRC50 for the tempering at 600°C. The hardness values obtained reflects the role of the martensitic matrix obtained after tempering at 250°C and 400°C.

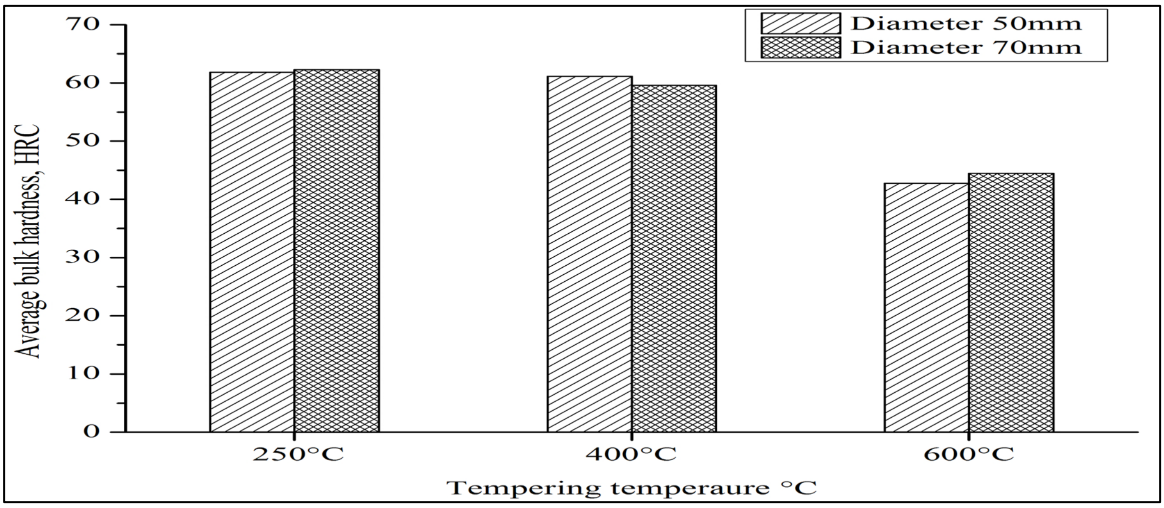


Figure 4: Average bulk hardness as a function of tempering temperatures of balls 50 and 70 mm in diameter

3.2 Optical Microscope Images and XRD Results

The excellent hardness obtained after tempering at 250°C and 400°C is following to the presence of a network of M_7C_3 eutectic carbides in a martensitic matrix in their microstructures (Figures 5a and 6a).

As shown in both Figures 5b and 6b, the diagrams of XRD obtained respectively on the 50 and 70 mm diameter balls tempering at 250°C, shows the presence of carbides type M_7C_3 distributed in a martensitic matrix. The residual austenite no transformed into martensite after quenching is also present.

The tempering at 600°C on the destabilized balls produced an important drop in the hardness due to the decomposition of martensite to a softer product (ferrite) (Figures 5c, 5d and 6c, 6d).

The diffraction spectrum (Figure 6b) of the ball of 70 mm in diameter reveals a higher level of residual austenite. This can be explained by the low cooling rate and the influence of the size of the balls which did not give the austenite sufficient time to transform completely, which led to the decrease of the martensitic transformation temperature Ms [19].

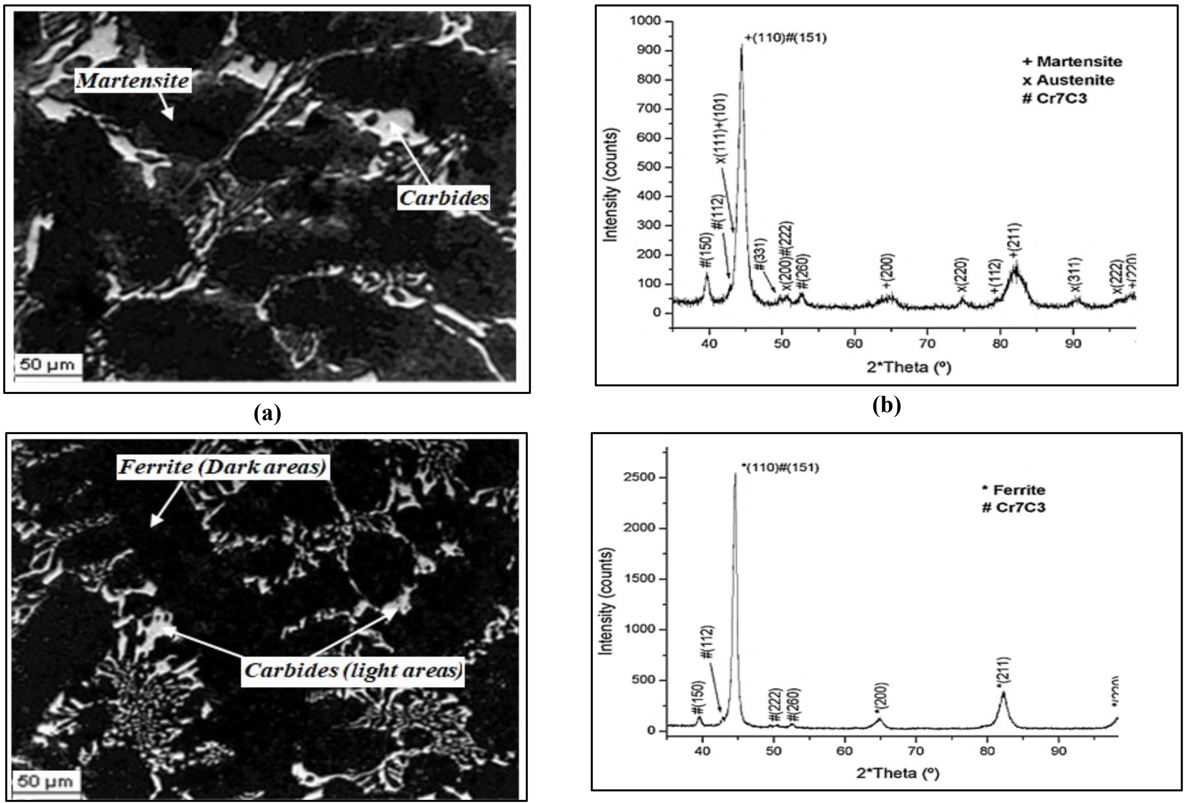
The microstructures showed a significant amount of eutectic carbides for 70 mm diameter balls that influence wear resistance.

3.3 Wear Results

The cumulative mass loss (%) of balls 50 and 70 mm in diameter, as-cast, and after tempering treatments is shown in Figure 7.

It is seen that wear resistance for balls at the as-cast state is quite good, but it is clearly improved after tempering at low temperatures (tempering at 250°C) and it is very poor after tempering at 600°C.

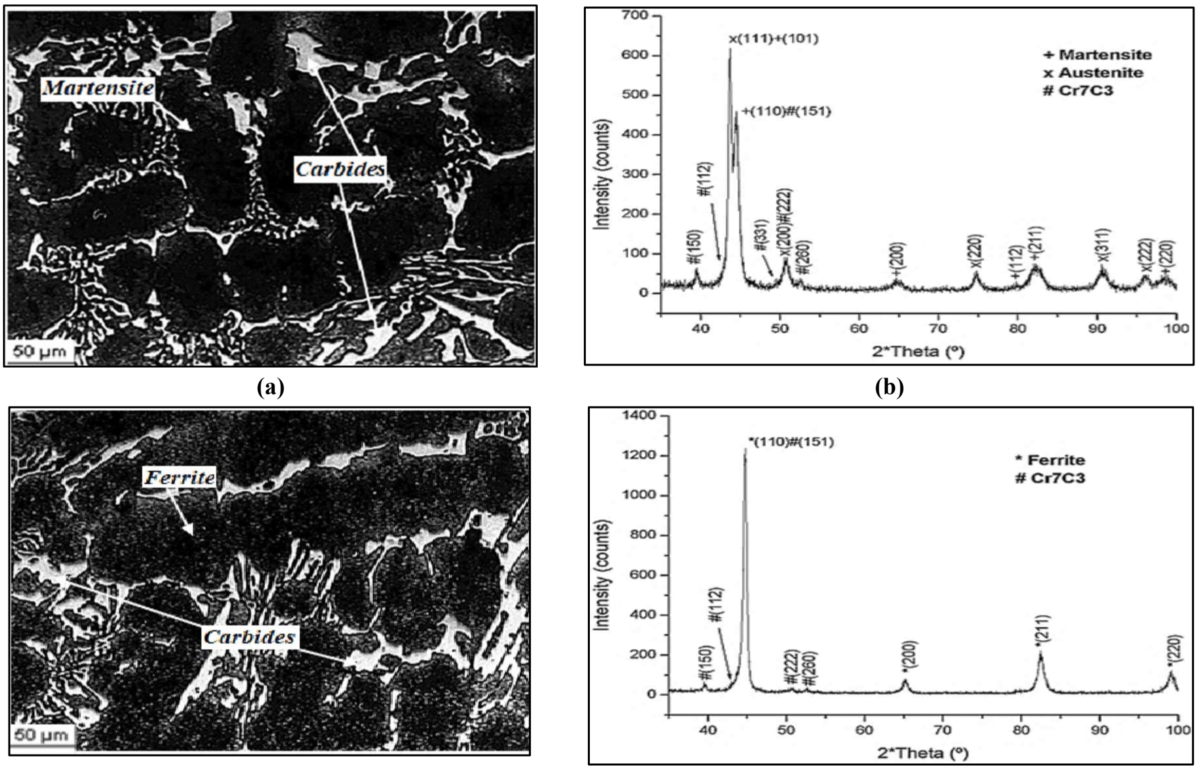
Carbides and the martensitic matrix, formed within the structure after tempering at 250°C, provide wear resistance to the cast austenitic structure. After the tempering at 600°C, the ferritic matrix formed, and most probably the carbides coarsening decreased the wear resistance balls [9]. The Effect of treatment tempering on hardness and abrasive wear of balls 50 and 70 mm in diameter is illustrated respectively in Figures 8 and 9.



(a) (b)

(c) (d)

Figure 5: Optical micrograph and X-ray diffraction pattern of 50 mm diameter grinding balls: (a, b) tempering at 250°C, (c, d) tempering at 600°C



(a) (b)

(c) (d)

Figure 6: Optical micrograph and X-ray diffraction pattern of 70 mm diameter grinding balls: (a, b) empering at 250°C, (c, d) tempering at 600°C

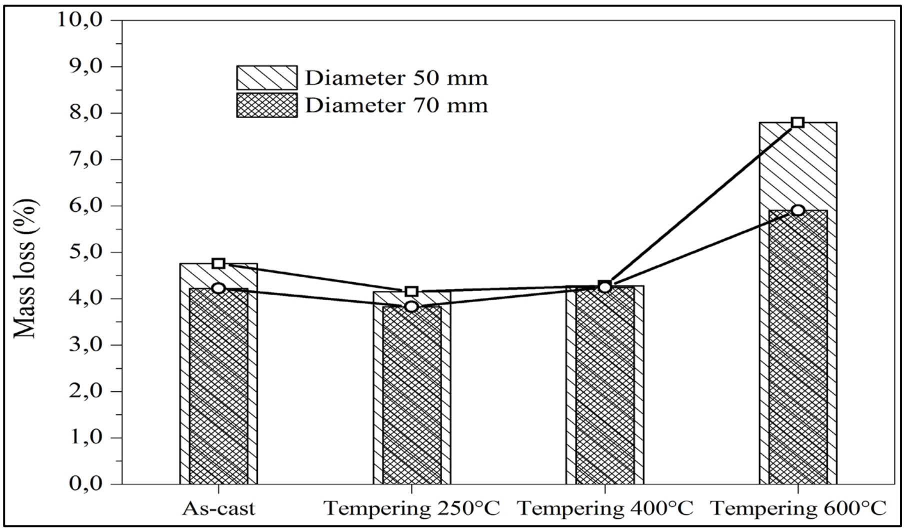


Figure 7: Effect of tempering treatments on wear of 50 and 70-mm diameter balls

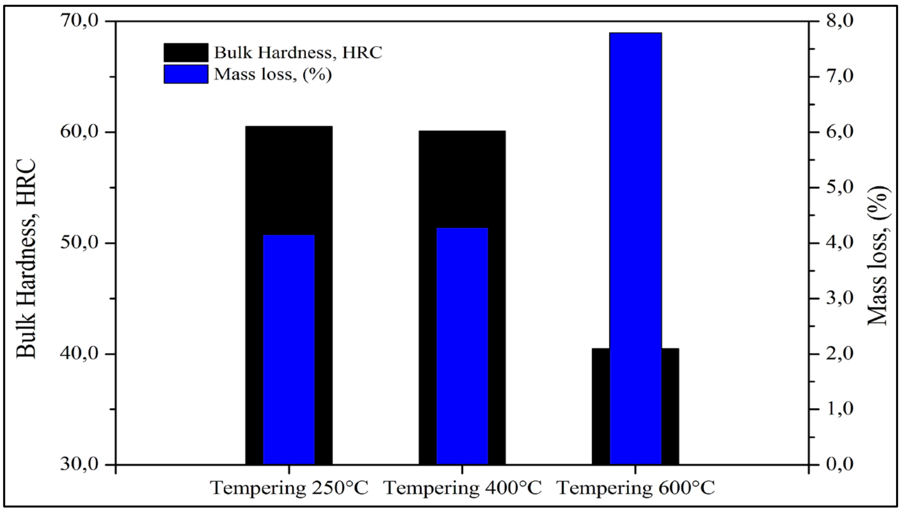


Figure 8: Tempering effect on hardness and abrasive wear of 50 mm diameter balls

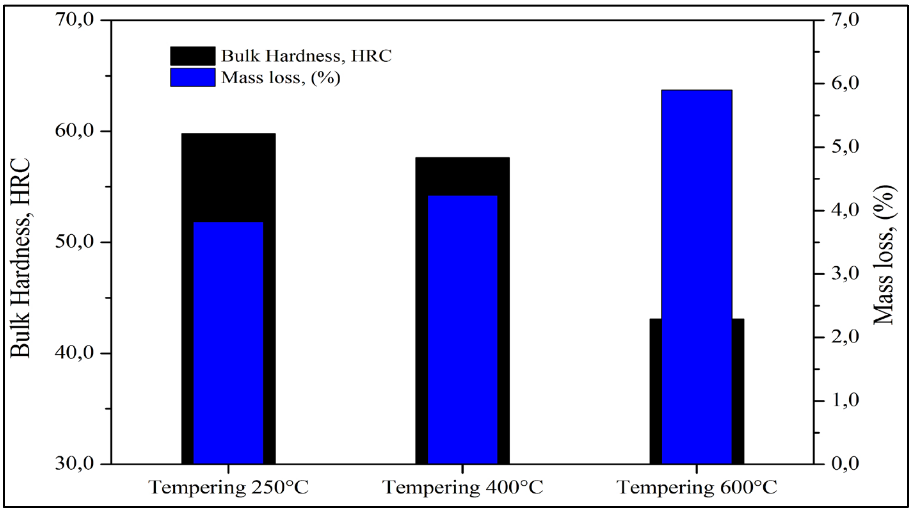


Figure 9: Tempering effect on hardness and abrasive wear of 70 mm diameter balls

The balls tempering at 250°C and 400°C, have a wear rate less important. This is explained by the value of the high hardness of these balls. The wear rate is most important for balls tempering at 600°C, which represents very low hardness.

It should be noted that the rate of wear of grinding balls 50mm in diameter is more significant than balls 70 mm in diameter, especially after high-temperature tempering (600°C), most probably connected with carbide component refinement in the first case.

4. Conclusion

The subcritical heat treatments (tempering) intended to cause relaxation of the stresses after quenching and reduces the risk of ball cracking is that of low temperatures, tempering at 250°C. The hardness after this tempering undergoes little variation compared to that obtained after quenching, whereas it decreases sharply after tempering at 600°C. It significantly improves the abrasive wear of high chromium white cast irons (HCWCI).

The hardness and wear resistance of high Cr cast iron depends on the type, amount of carbides, and matrix structure [7]. The martensitic matrix and secondary carbides precipitated during destabilization heat treatment as well as carbides formed during tempering at 250°C have played an important role in improving wear resistance and hardness.

The carbide morphology also influences the wear behavior of high chromium white cast irons. Cast irons with fine carbides and martensitic matrix are the greatest abrasive wear resistance.

The effect of different holding times at quenching temperatures and cooling mediums on the properties of high chromium cast irons will be studied later.

Acknowledgments

The authors are grateful for the support and valuable help of all laboratory staff and managers of the Algerian Foundries of Tiaret (ALFET)/Algeria.

Author contribution

All authors contributed equally to this work.

Funding

This research received no external funding.

Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of Interest

The authors declare no conflict of interest.

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